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[Note: Very poor copy of the original. Names, addresses, company names and brand names are translated in the most common manner. Japanese language does not have singular or plural words unless otherwise specified by a numeral prefix or a general form of plurality suffix.]

Description of the Invention**1. Name of the Invention****Manufacturing Method for High Melt Point Glass Body****2. Scope of the Claims**

Manufacturing method for the preparation of high melt point glass body characterized by the fact that a sintered body from a mixed material that is an Al₂O₃ – Ln₂O₃ system (where Ln represents rare earth metal element and yttrium element), which is difficult to form a glass state, and which is formed as relative to the fine powder material of α-Al₂O₃, one type or two or more types of any Ln₂O₃ fine powder materials, are added, is heated at a temperature of approximately 2500oC or higher, and preferably at a temperature of 3000oC or higher using an arc plasma flame, and it is melted, and this is then rapidly cooled and a transparent to visible light beam ceramics glass body is obtained continuously.

3. Detailed Description of the Invention

The present invention is an invention about a large scale manufacturing method where a high melting point oxide material, which is difficult to form a glass state, and its system, are melted by using an arc plasma flame and this material is supplied in the gap between cooling rolls that are rotating at a high speed, and it is rapidly cooled and it becomes a material in a glass state, and a ceramic glass body that is transparent to visible light is obtained.

Among the many oxide compounds, as it is well known, as the components that easily form a glass state there are B₂O₃, SiO₂, GeO₂, P₂O₅, As₂O₅, etc. The present invention is an invention whereby relative to this, improves the rapid cooling methods used according to the previous technology relative to the oxide compounds and their systems, which are difficult to form a glass state, like for example, Al₂O₃ – Ln₂O₃ (where Ln represents rare earth metal element and yttrium element), and it uses an arc plasma flame and an impact quenching etc., high speed cooling method, and it realizes a new Al-Ln-O glass state.

Namely, it is an invention that suggests a manufacturing method for the preparation of a glass body from an Al₂O₃ – Ln₂O₃ system (where Ln represents rare earth metal

element and yttrium element), which has been said to be difficult to form a glass state according to the previous technology, and according to the present invention, first a sintered body which is formed as relative to the fine powder material of α -Al₂O₃, one type or two or more types of any Ln₂O₃ fine powder materials, are added, is heated at a temperature of approximately 2500°C or higher, and preferably at a temperature of 3000°C or higher using an arc plasma flame, and it is melted, and this is then rapidly cooled, for example by the method where it is supplied in the gap between cooling rolls rotating at a high speed, and a transparent to visible light beam ceramics glass body is obtained continuously.

Here below, an explanation will be provided relative to the manufacturing of Al₂O₃ - Ln₂O₃ system glass body.

Granulated below 325 mesh (45 microns), fine powder form, high melting point oxides of α -Al₂O₃ and Ln₂O₃ were mixed at different mole ratios, and sintered bodies with a cylindrical shape with dimensions of 3 mm diameter x 30 mm, were formed. This sintered bodies were placed in a chuck and their edges were melted by a two stand arc plasma flame and the molten material flowed in the gap between two rotating at a high speed rollers of an inner part cooling device and by that it was possible to produce a transparent to the visible light experimental material with a thickness of approximately 1 micron and a diameter of approximately 50 mm. Regarding the mole ratio of the α -Al₂O₃ and the Ln₂O₃ in this case, it is preferred that the ratio of the Ln₂O₃ relative to 1 mole of α -Al₂O₃ be within the range of 0.1 ~ 10 moles. Naturally, when both materials are used individually a glass body is not obtained. The fact whether or not the obtained by this method experimental material is a glass material was studied by using a polarized light microscope, an X-Ray diffraction and an electron microscope. According to the method using a polarized light microscope, the experimental material was placed in the space between orthogonal Nicol and an orthoscopic observation was conducted. For the experimental material, even if the stage was rotated, a change in the image contrast was not observed. Then, for the X ray diffraction image and for the electron beam diffraction image, only a halo image was observed. In the viewing field by the electron microscope there was no intervening material observed. In Figure 1 the electron beam diffraction image (Figure 1 - 1) of the experimental material from the Al-Ln-O system and its planar viewing field image (Figure 1 - 2), are presented. The phenomenon of crystallization of the Al-Ln-O system experimental material by subjecting it to a thermal treatment at a temperature of 1000°C for different number of hours was studied by using X ray diffraction. The results from that are shown in Figure 2. From the above-described observations it is possible to determine that the experimental material obtained by using the above-described equipment is a glass material. Regarding such glass material, it is possible to obtain various compositions of the Al-Ln-O system, and the elements that are represented by the above described Ln are La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu and Y. Regarding the produced glass material, it is transparent relative to visible light, and also, regarding the Ln element, usually, the elements that are present in a third valency are stable, however, among the Al-Ln-O glass materials, the materials where Ln is Sm, Eu and Yb and these elements are present in a bivalent state, it is

considered that a coloration is developed. In Figure 3 the obtained glass material is presented.

The coloration of the obtained Ln-Al-O system glass is according to the described here below.

<u>Ln-Al-O</u>	Color
La-Al-O	colorless
Ce-Al-O	colorless
Pr-Al-O	pale green color
Nd-Al-O	pale blue color
Sm-Al-O	brown color
Eu-Al-O	pale yellow color
Gd-Al-O	colorless
Tb-Al-O	colorless
Dy-Al-O	colorless
Ho-Al-O	colorless
Er-Al-O	pale orange color
Tm-Al-O	colorless
Yb-Al-O	pale brown color
Lu-Al-O	colorless
Y-Al-O	colorless

Regarding the glass materials that is obtained by using the above described glass material manufacturing installation, and using an oxide material or its system that are difficult to form a glass state irrespective of the type of the used Al-Ln-O system, it is anticipated that they are materials that have properties that are different from those of the glass materials obtained according to the previous technology from glass, B₂O₃, SiO₂, etc., and it is considered that from the standpoint of the optical, electric and magnetic properties, they are materials that can play an extremely important role in the different aspects of the electronic memory related technologies and also in other processing technologies.

Practical Examples

The manufacturing of high melting point ceramic glass materials uses the equipment presented according to Figure 4. Here below an explanation will be provided by using the figure.

In the figure, 1 represents a chuck whereby in order to produce the glass material, the sintered body experimental material can be moved in the up and down direction within the diagram. Also, in the figure, 2 represents the sintered rod. The material used in order to obtain the glass material, is a material where less than 325 mesh dispersity, fine powder form α -Al₂O₃ and Ln₂O₃, for example, La₂O₃, powder are weighed at the

corresponding mole ratio, and after that these are well mixed and combined by using a mixing device, and this material is press molded in a cylindrical shape with dimensions of 3 mm diameter x 50 mm, and this cylinder shape material is sintered at a temperature of approximately 1000°C for a period of 20 hours in an air atmosphere. The cylinder shaped sintered material body 2 is grasped by the chuck 1 so that, as shown according to the presented in Figure 1, its front end is introduced into an arc plasma flame. 3 represents argon arc plasma flame (with a temperature of at or above approximately 3000°C), and it is at a temperature of approximately 2500°C or above, and preferably, it is at or above approximately 3000°C. 4 represents the arc plasma nozzle, 5 represents the roller where the inner part is cooled by water, and that rotates at 1000 rpm or higher, and where by the motion in the left and right direction, it is possible to adjust the thickness of the glass material. The molten material obtained from the sintered body enters in the gap between the two rollers that are rotating at a speed of approximately 1000 rpm, and from the rollers, a transparent glass material with a thickness of approximately 1 micron, is obtained. The obtained glass material has a diameter in the range of 50 ~ 100 mm. Moreover, the details of the cooling part are shown in Figure 5. 6 (in Figure 4) represents the experimental material controlling device, 7 represents the produced glass material. This glass material is collected in the receptacle tray 8.

In Figure 5, 9 represents the motor used for the rotation, 10 represents the entrance in the cooling part where the cooling part used cooling water is transported, 11 represents its exit opening. The cooling water enters through the above described opening 10 close to the roller inside part separation wall 12 and it cools the roller surface. The water that has a somewhat higher temperature is directed to exit through the exit opening 11 by 13, which is close to the axis part.

Moreover, in Figure 6, a schematic diagram is shown of the essential parts of the device generating the above described argon arc plasma. If we are to provide a simple description, through the protection gas nozzle 14, as a protective gas 15, for example, a mixed gas containing 93 volume % Ar and 7 volume % H₂ is used. 16 represents the melt injection head, 17 represents the cooling water. 18 represents the plasma gas (Ar), 19 represents the tungsten electrode (- electrode), 20 represents a high frequency wave, 21 represents the electric source for the pilot arc, 23 represents the electric source for the melt injection arc. 23 represents a switch, 24 represents an arc plasma flame, 25 represents a (+electrode).

After that, the glass material that is obtained by using this equipment is presented in Figure 3.

In the case of this glass material, it can be obtained from all rare earth type elements and yttrium element (Y) and also, it can be obtained from almost all the mole ratios of the α-Al₂O₃ and Ln₂O₃, however, it is preferred that relative to 1 mole of the α-Al₂O₃, the amount of the Ln₂O₃ is within the range of 0.1 ~ 10 moles. The confirmation of the glass state of the material was conducted by using polarized light microscope, X ray diffraction and electron beam diffraction.

In the above described Figure 1, the electron beam diffraction pattern and the microscopic image of the glass material obtained as Al₂O₃: Ln₂O₃ = 6:1 are weighed, as a representative example of the Al-Ln-O system, are shown. For the electron microscope a manufactured by Nippon Denko Company, 200 kV microscope, was used. Regarding the electron beam diffraction image, it was projected at an acceleration electric potential of 150 kV, and it showed a typical halo image. The fact that this halo image was obtained indicates that the obtained experimental material is a glass material. Regarding the electron microscopic image, it is an image obtained by a bright viewing field image at a magnification of 62,000 times. From this image it is seen that there are no intervening materials present at all and this indicates that the obtained glass material is a microscopically good glass material. Then, by the observation through a polarized light microscope, it is confirmed that even when the experimental material is rotated, there is no change in the contrast at all, and this indicates that macroscopically also it is a good glass material. Also, in Figure 2, the results are shown from a measurement conducted by an X-ray diffractometer using CuK α relative to the manufactured glass material after it has been subjected to a thermal treatment for the time period as shown in the figure, and this studies the conditions of the crystallization.

As it has been described here above, according to the present invention it is possible to suggest a manufacturing method for the preparation of high melt point glass body characterized by the fact that a sintered body from a mixed material that is an Al₂O₃ - Ln₂O₃ system (where Ln represents rare earth metal element and yttrium element), which is difficult to form a glass state, and which is formed as relative to the fine powder material of α -Al₂O₃, one type or two or more types of any Ln₂O₃ fine powder materials, are added, is heated at a temperature of approximately 2500°C or higher, and preferably at a temperature of 3000°C or higher using an arc plasma flame, and it is melted, and this is then rapidly cooled by using for example a method where this molten material is rapidly cooled in the space between rotating at a high speed cooling rollers and a transparent to visible light beam ceramics glass body is obtained continuously.

Here above, mainly, a practical example was described where La₂O₃ was used as the Ln₂O₃, and also, as the rapid cooling method for the material that has been melted by the argon arc plasma, water cooled type, high-speed rotating rollers were used, however, after this, as other practical example, there is the example where Nd₂O₃ was used as the Ln₂O₃, and where for the rapid cooling method, the equipment shown according to Figure 7, that has a structure formed from a water cooled piston 26 and an anvil 27, was used.

Regarding the α -Al₂O₃ and Nd₂O₃ that are used as the material, they are both materials where the purity level is at least 99.9 % or higher, and also, they are materials that are in a fine powder form. The mole ratio of both materials, namely, α -Al₂O₃:Nd₂O₃ = x : 1, where x was within the range of 1 and 10. Both materials were well pulverized, mixed and combined, and they were subjected to an elevated pressure of 4 ton/cm², and pellets with a thickness of 1 mm and a diameter of 5 mm, were formed. These pellets were sintered in an air atmosphere at a temperature of 1000°C for a period of 5 hours. The pellets 28 of this sintered experimental material were placed inside a manufactured from

Cu piston, as shown according to Figure 7, and they were melted by the plasma flame 25 until the experimental material formed a spherical shape. While heating by using the plasma flame 25, the water cooled by the cooling water 30 piston 26 and the manufactured from copper anvil 27 are operated by the spring 31 and the electro-magnet (not shown in the figure), and the molten material is enclosed in the space between the two and it is rapidly cooled. Moreover, in this case, the above described plasma flame 25 is discharged from the plasma torch 32.

Regarding the produced glass material, at a diameter of approximately 5 mm and a thickness of approximately 1 micron, it is a material that is transparent to visible light beam. The glass material obtained from the α -Al₂O₃ : Nd₂O₃ = 6 : 1 experimental material was subjected to a an orthoscopic observation by the polarized light microscopic method, in the space between orthogonal Nicol, and the same way as in the above described practical example, even if the stage was rotated, there was no change in the image contrast. Then, through the X ray diffraction pattern, and the electron beam diffraction image, only a halo pattern was observed. Then, when using an electron microscope, in the bright viewing field image there were no intervening materials observed. Figure 8 is a diagram presenting the results from the X ray diffraction studies of the crystallization phenomenon in the case when the above described Al-Nd-O system experimental material was annealed at a temperature of 1000°C for different number of hours (CuK α radiation, (using Ni filter), pulse height analysis).

From the above described it is confirmed that the isotropic properties possessing materials that are obtained from the 6 α -Al₂O₃. Nd₂O₃ obtained from each of the above described experimental materials, are glass materials.

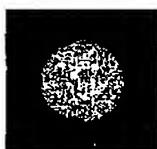
4. Brief Explanation of the Figures

Figure 1-1 represents the electron beam diffraction pattern (150 KV) of the Al-La-O type glass material; Figure 1-2 represents its bright viewing field pattern (x 62500); Figure 2 represents the results from the measurement of the crystallization of the Al-La-O type glass by the X ray diffraction method. Figure 3 represents a photograph of a thin piece of the Al-Ln-O type glass material. Figure 4 represents the glass material manufacturing equipment according to the first practical example of the present invention. Figure 5 represents a front view diagram where one part of the inner part of the cooling roller 5 from Figure 4, has been cut open. Figure 6 represents a schematic diagram showing the essential parts of the argon arc plasma generating equipment according to the present invention. Figure 7 is a glass manufacturing equipment related to another practical implementation example according to the present invention. Figure 8 is a line chart diagram showing the results from the X ray diffraction measurements of the crystallization of the same Al-Nd-O type glass.

- 1.....chuck for the sintered body of the experimental material,
- 2.....sintered rod, 3.....argon arc plasma flame,
- 4.....arc plasma nozzle, 5.....cooling roller,
- 6.....experimental material controlling device,

- 7.....synthesized glass material, 8.....glass material
receptacle tray, 9.....motor, 10.....cooling water entrance
opening, 11.....cooling water exit opening,
12.....inner part perimeter vicinity, 13.....inner part
axis vicinity, 14.....protective gas nozzle,
15.....protective gas, 16.....discharge
head, 17.....cooling water, 18.....plasma gas (Ar),
19.....tungsten electrode (- electrode), 20.....high
frequency, 21.....electric source for the pilot arc,
22.....electric source for the melt radiation arc,
23.....switch, 24.....arc plasma flame,
25.....roller (+ electrode), 26.....piston,
27.....anvil, 28.....pellets, 29.....plasma flame,
30.....cooling water, 31.....spring,
32.....plasma torch.

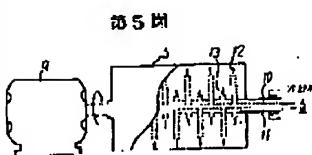
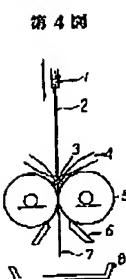
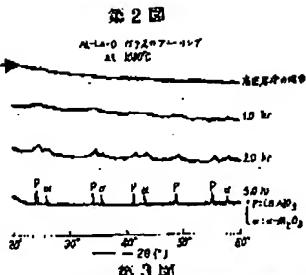
第1圖-1



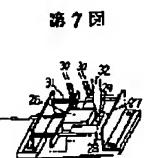
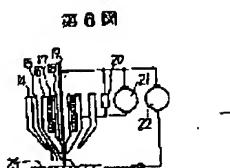
第1圖-2



In the case of high frequency

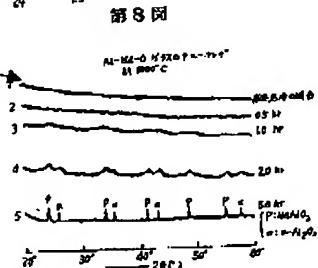


In the case of high frequency



九、對財務類中印件	
六月	四
七月	六
八月	四
九月	三

8. 本定は外の把頭告、特許出願入主たば北界
並其見解



國上中內外四境之說
或謂中國之內爲中國，中國之外爲外國，此非也。中國者，我
國之謂也。中國之內爲我國，中國之外爲他國，此是也。

5. Record of the Appended documents

(1) Description	1 copy
(2) Figures	1 copy
(3) Application copy	1 original
(4) Power of attorney	1 copy

**6. Other than the above described invention authors, patent applicants or
representatives****Patent Assignee: (Toho) Research Institute Iron and Steel***Translated by Albena Blagev ((651) 735-1461 (h), (651) 704-7946 (w))**12/07/04*

卷之三

頤 許 特

特許厅長官　山　谷　一　殿

1. 発明の名称

セラミックスのガラス体の製造方法

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四
五
六

1. 発明の名称 高散点セラミックスのガラス体
の製造方法

上神許前家の鏡面

ガラス状態になりにくく、 $\text{Li}_2\text{Al}_2\text{O}_5 \cdot \text{In}_2\text{O}_3$ 系と似た
し In_2O_3 は耐熱強元素およびイソトリウム元素を示す
の高融点酸化物において摩耗試験 $\text{U}-\text{Al}_2\text{O}_5$ に對し
 In_2O_3 の何れかノ複又は2種以上から成る複合材
を加えて成る複合物の磨耗体をアーケアクスズマフ
レームにて約3500°C以上好ましくは約5000°C以上
に加熱熟成せしめ、これを高速急冷せしめ可燃
光素にて透明なセラミクスガラス体を逐段的に
得ることを特徴とする高融点セラミクスガラス
体の製造方法。

上場の詳細を説明

本発明はガラス状態になりにくい高融点化物及びその系をアーチアラズマフレームにより溶融し、之を高速回転する冷却ロール間に接触し、高速冷却してガラス状態にし、可視光線に対して透明なセラミックガラス体を大量に製造する方法

⑯ 日本国特許庁
公開特許公報

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⑬出願日 昭48(1973) 7. 6
審査請求 未請求 (全5頁)

序內整理番号

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7417 41

日本分類
200XC/29
21 A2

⑤ Int.CP²
COPC 3/12

既に開てみた日本でも

多くの酸化物の中でガラス状態になりやすい骨格成分は、層状のようす Ba_2O_3 、 Ba_1O_2 、 GeO_2 、 P_2O_5 、 La_2O_3 等である。本実験はこれに反し從来からガラス状態になり難いとされていた酸化物及びその系、例えば $\text{Al}_2\text{O}_3 - \text{Zn}_2\text{O}_3$ 系 (Zn は軽土類元素群などびツトリウム元素をさす) を、従来の急冷方法を改良し、ターゲットラズマフレームとインパクトエンテンディング等の高速急冷法を使用して、初めて $\text{Al}_2\text{O}_3 - \text{Zn}_2\text{O}_3$ 系のガラス状態を達成したのである。

すなわち、延伸からガラス状態になりにくくと
示せられていた $\text{Li}_{2}\text{O} - \text{SiO}_2$ 系(ここで LiO は
角土鉱元素かよびイソトリウム元素を示す)のガ
ラス体の脆性を万能化を提供しようとするものであり、
本説明では先づ、慣例的 $\text{Li}-\text{Al}_2\text{O}_3$ に如し $\text{Li}_2\text{O}-\text{SiO}_2$ の
何れか! 他又は 2 個以上の酸粉末を加えてなる混
合物の焼結体をアーチアラズマフレームにて約
2500 °C 以上好ましくは約 3000 °C 以上で加熱融解
せしめ、これを弱れば高速度冷却却ホール面内で

熱をせしめる如き高遡率の方法によつて処理せしめ、可燃光輝にて透明なセラミックガラス体を半硬的に得られるようになつものである。

以下 $\text{Al}_2\text{O}_3 - \text{Zn}_2\text{O}_3$ 系のガラス体固溶物に関する説明を行なう。

ZrO_2 マンシュー (M.M.) 以下の脱皮にした脱皮純度脱離化物 (Al_2O_3 やおよび Zn_2O_3) を各所のモル比で混合し、 $900^\circ\text{C} \times 24\text{hr}$ の円柱状の焼結体にした。この焼結体をチャックで用ひし、その先端を 2 台のアーチプラズマフレームで溶解し、被界物を内焰冷却装置のついた高遡回転している 2 台のローラー間に流しこむことによつておのずの間引きを有するための内焰冷却装置の外側に外焰冷却装置の外側ではガラス体は得られていねい。このようにして得られた試料がガラス体であるかどうかは偏光顕微鏡、X 線・回折及び電子顕微鏡によつて調べた。偏光顕微鏡を用いる方法では直交

特開昭50-25808(2)
クロム間に試料を置き、オルソスコープ観察を行なつた。試料のかいてあるステージを回転してもそのコントラストに変化は観察されなかつた。さらに X 線回折像、電子顕微鏡ではロー像しか觀察できなかつた。電子顕微鏡による明視野像では外在性は觀察されなかつた。一方他の $\text{Al}_2\text{O}_3 - \text{Zn}_2\text{O}_3$ 系における試料の電子顕微鏡像 (図 1 図 2) 及びその回折像 (図 3 図 4) を示す。 $\text{Al}-\text{Zn}-\text{O}$ 系の脱皮を 1000°C でいろいろな時間熱処理することによつて結晶化する現象を X 線回折で調べた。その結果を表 2 図に示す。以上の观察より上記の脱皮で得られた試料はガラス体であることが何足らず。このようなガラス体は $\text{Al}-\text{Zn}-\text{O}$ 系のあらゆる組成のところで得られた。得られた元素は $\text{La}, \text{Ce}, \text{Pr}, \text{Nd}, \text{Tm}, \text{Bn}, \text{Eu}, \text{Gd}, \text{Dy}, \text{Ho}, \text{Er}, \text{Tb}, \text{Lu}$ 及びすである。作成したガラス体は可燃光輝に対して透明であり、又 2 种元素は一般には 2 値で存在するのが定であるが $\text{Al}-\text{Zn}-\text{O}$ 系ガラス体の中で Ln が 3 値、及び Dy ではそれらの元素が 3 値で存在している。

と思われる色彩を呈している。第 2 図に得られたガラス体を示す。

得られた $\text{Zr}-\text{Al}-\text{O}$ 系の透明なガラスの色彩は次の如くであつた。

組成	色
$\text{La}-\text{Al}-\text{O}$	無色
$\text{Ce}-\text{Al}-\text{O}$	無色
$\text{Pr}-\text{Al}-\text{O}$	薄い緑色
$\text{Nd}-\text{Al}-\text{O}$	薄い青色
$\text{Tm}-\text{Al}-\text{O}$	桃色
$\text{Bn}-\text{Al}-\text{O}$	淡黄色
$\text{Gd}-\text{Al}-\text{O}$	無色
$\text{Dy}-\text{Al}-\text{O}$	無色
$\text{Ho}-\text{Al}-\text{O}$	無色
$\text{Er}-\text{Al}-\text{O}$	薄い緑色
$\text{Tb}-\text{Al}-\text{O}$	無色
$\text{Lu}-\text{Al}-\text{O}$	薄い褐色
$\text{Y}-\text{Al}-\text{O}$	無色

上記のガラス体強度測定を使用して $\text{Al}-\text{Zn}-\text{O}$ 系のみならずガラス状態になり難い複合物及びその系において得られるガラス体は通常のガラス、 B_2O_3 、 SiO_2 等の系よりなるガラスとは異なつて特徴を持つことが予想され、光学的、物理的、電気的性質の立場から電荷子関係其の他工芸的の点各方面で非常に役立つものと思われる。

実験

本説明セラミックガラス体強度は前半に示す装置を使用する。以下四図を用いて説明を行なう。

1 はガラス体を作成する為の焼結体試料チャックで途中で上下に動作できる。2 は焼結体を示す。ガラス体を得る為の試験機、3 は ZrO_2 以外の脱皮にした脱皮純度脱離化物 (Al_2O_3 と Zn_2O_3 例えば Zn_2O_3) の粉末を適当なモル比に秤量した後、搅拌器でよく混じし $900^\circ\text{C} \times 24\text{hr}$ の円柱状にプレス成形した。この円柱状物質を約 1000°C で約 2 時間大気中で焼結したものがである。円柱状焼結体 3 を図 1 図 2 示すようなチャック 1 に挿み、先端がアルゴンター

アラズマフレームの中に入るよう設置する。これはアルゴンアーケタラズマフレーム（約2000°C以上熱温度）を示し、約2500°C以上、好ましくは約3000°C以上である。これはアーケタラズマノズルを示す、これは水で内冷却してあるローラーを示し、1000 rpm以上で回転し、左右に移動することができるガラス体の厚さを範囲できる。總結体が導出した瞬間約1000 rpmの速度で回転している場合のローラーの間に人りローラーからは約1ミリの厚さを持つ透明なガラス体が得られた。得られたガラス体は直角約45°～100度の大きさを有している。

なお、冷却槽の詳細については図を図示す。これは試料のかきとり器を示すこれは作成したガラス体を示す。これをガラス体の脱臼によって受け取る。

図2の左は回転駆動用モーターを示し、右は冷却水分配冷却装置は冷却水を流入入口を、右はその出口を示す。冷却水は節理入口10から入りローラー内部の距離近くに入りローラー表面を冷却する。

前記系/図にはAl-Zn-O系の典型的な例として $Al_2O_3 : Zn_2O_3 = 4 : 1$ に算定して得られたガラス体の電子顕微鏡像および断面観像が示してある。使用した電子顕微鏡は日本電子社製の300 kVのものである。電子顕微鏡像は加速度圧130 kVで撮影し、典型的なハロー像を示している。このハローから得られた試料がガラス体であることを示している。電子顕微鏡像は明視野像で62,500倍のものである。この像からこのガラス体には全く介在物が存在せず複雑な組織のガラス体であることを示している。さらに電子顕微鏡による観察で試料を回転してもコントラスト変化が全くないことから巨視的にも均一のガラス体であることを示している。また、図2には、観察したガラス体を測定するよう瞬間熱遮蔽した後、0.02mm幅によるX線ダイフラクトメータによって測定した結果であり、結晶化の様子を調べたものである。

以上述べたように、本発明によれば、ガラス状態になりにくい $Al_2O_3 - Zn_2O_3$ 系（固相反応は総合元素およびイットリウム元素（Y）を示す）の

特開昭50-256083)す。若干過度の上つた水を輪形の近くよりから出ロ11へ出す。

なお、図6は同記のアルゴンアーケタラズマ焼成装置の更細の模式図を示す。簡単に説明するが、本装置はガスノズルで、保護ガスなどとしては、例えば、Ar 93%質量、H₂ 7%質量の混合ガスを使用する。14は燃焼ヘッドであり、15はその冷却水である。16はラズマガス（Ar）、17はラングスキシング燃焼（一眼炉）。18は高周波、19がバイオラートアーケーのための試料、20が溶融アーケーのための電磁石を示す。21はスイッチ、22はアーケタラズマフレーム、23はローラー（+管架）を示す。

次に図2の鏡像を用いて得られたガラス体の写真を示してある。

このガラス体は総合元素のすべておよびイットリウム元素（Y）で構成され、また Al_2O_3 と Zn_2O_3 の殆んどすべてのモル比のところで得られ、好ましくは Al_2O_3 / モルに對し Zn_2O_3 0.1～10モルである。ガラス体であることとの同定は偏光顕微鏡、X線回折、電子顕微鏡によつて行なつた。

高能点焼化物において微弱状 Al_2O_3 に対 Zn_2O_3 の何れか／又は2種以上から成る微弱末を加えて取る混合物の混和体をアーケタラズマフレームにて約2500°C以上好ましくは約3000°C以上に加热溶解せしめ、これを高周波回転角ローラーにて加热せしめる等の急冷方法を用いることによつて、可視光域にて透明なセラミックガラス体を典型的に得る高融点セラミックのガラス体回収率万倍を達成することができる。

以上主として Zn_2O_3 として Zn_2O_3 を取り上げ、かつアルゴンアーケタラズマによる熔融体の最高温度として、水冷式高周波回転ローラーを採用した実験について述べたが、次にさらに他の実験として Al_2O_3 として Al_2O_3 を用い、急冷方法として第7回に示す如き水冷されたピストンなどから噴出された液滴を採用した。

料として用いる Al_2O_3 および Na_2O_3 は純度純度として99.9%以上のものであり、また微弱末を用いる。両者のモル比、すなわち Al_2O_3 ：

$\text{Ba}_{2}\text{O}_3 \cdot x$: / シズはノと10の間の範囲にある。内容をよく粉砕混合しへ $300\text{kg}/\text{m}^3$ に加圧し、厚さノ・1mm程度のペレットを形成する。このペレットを脱気中にて 1000°C にて焼成焼結する。この焼結された試料のペレットを 2°C 図に示す40個のピストンの中央部までアラズマフレームが環状にかかるまでアラズマフレームがによって加熱される。アラズマフレームがによって加熱されながら冷却水がによつて冷却されるピストンがと匣型のかなと (アンビル) ととをスプリッピングと可燃石 (図示せず) で所定させ、尚且の間に均して常温試料を盒用に構成せよ。なお、この場合アラズマフレームはアラズママートーチはから放出される。

生成したガラス体は匣型約 3cm 厚さ約 1cm で可視光版で透明である。 $\text{Al}_2\text{O}_3 \cdot \text{Ba}_{2}\text{O}_3 \cdot x$: / の試料について焼成されたガラス体を偏光顕微鏡の方法で観察にてオルソスコープ観察を行つた。前記実施例におけると同様にステージを回転しても率のコントラストに変化はなかつた。以下に工場分析を、電子顕微鏡像ではハローをしか

識別できなかつた。さらには電子顕微鏡による明視野像では分在物は識別されなかつた。第1图は前記の $\text{Al}-\text{Ba}-\text{O}$ 系の試料ガラス体を 1000°C で約々の時間アニーリングすることによつて結晶化する現象を电解回折で調べた結果である (0.016波長 (1.17Å) 使用。パルスの厚さ四分の一)。以上の各試料から $\text{Ba}_{2}\text{O}_3 \cdot \text{Ba}_{2}\text{O}_5$ から焼成された萬能均性ガラス体であることが同見された。

4. 四圖の圖解を附明

第1图-1は $\text{Al}-\text{La}-\text{O}$ 系ガラス体の電子顕微鏡像 (100KV)。第1图-2はその明視野像 ($\times 42500$)。第3图は $\text{Al}-\text{Ba}-\text{O}$ 系ガラスの結晶化のX線回折法による測定結果。第4图は $\text{Al}-\text{La}-\text{O}$ 系ガラスの薄片の写真。第5图は本発明の一実施例に供するガラス体無遮蔽像、第6图は第5图の暗箱ローラー-1の内面を一部切削して示す近屈図。第6图は本発明に供するアルゴンシーカークアラズマ丸棒装置の前面をボックス式図、第7图は本発明の他の実施例に供するガラス体無遮蔽像、第8图は同じく $\text{Al}-\text{La}-\text{O}$ 系ガラスの結晶化のX線回折法による測定結果。

来を示す図である。

1...塊體試料テナクト、2...規範標、3...アルゴンシーカークアラズマフレーム、4...シーカークアラズマノズル、5...冷却ローラー、6...試料かきとり筒、7...生産ガラス体、8...ガラス体裏面、9...モーター、10...冷却水入口、11...冷却水出口、12...内周周縁附近、13...内底軸附近、14...保護ガスノズル、15...導管ガス、16...密封ヘッド、17...冷却水、18...アラズマガス (Ar)、19...タンクシステム電源 (抵抗)、20...高周波、21...バイロクトアークのための制限、22...舟載テナクトのための凹版、23...スイッチ、24...シーカークアラズマフレーム、25...ローラー (+ 軸承)、26...ピストン、27...かなと C (アンビル)、28...ペレット、29...アラズマフレーム、30...冷却水、31...スプリング、32...アラズママートーチ。

第1图-1

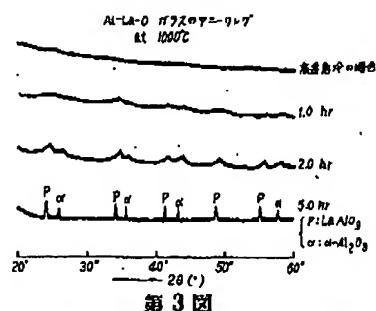


第1图-2



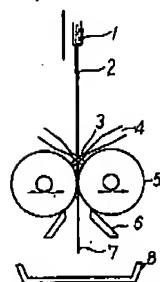
特許 第50-25608(S)

第2図

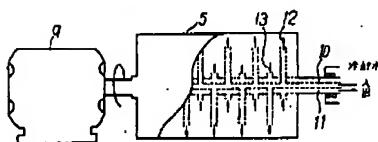


第3図

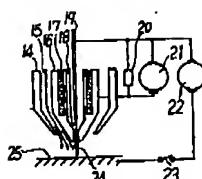
第4図



第5図

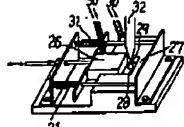


第6図



第8図

第7図



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